

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

WASHINGTON, D.C. 20460

OFFICE OF  
PREVENTION, PESTICIDES  
AND TOXIC SUBSTANCES

September 25, 2007

**MEMORANDUM**

SUBJECT: Review of "Measurement of Transfer of Deltamethrin Residues from Vinyl and Carpet Flooring Treated with a Fogger Formulation as a Function of Time"  
(MRID#: 462976-03) D336775.

FROM: Matthew Lloyd, Industrial Hygienist  
Reregistration Branch 1  
Health Effects Division (7509P)



THROUGH: Jeff Evans, Biologist  
Chemistry and Exposure Branch  
Health Effects Division (7509P)



TO: Cathryn O'Connell  
Reregistration Branch 2  
Special Review and Reregistration Division (SRRD) (7508P)

Attached is a review of a study conducted to determine the transfer of Deltamethrin (DTM) residues from vinyl and carpet flooring from the application of an indoor aerosol fogger. The study was submitted by the Pyrethrin, Piperonyl Butoxide, MGK-264, Deltamethrin, Non-Dietary Exposure Task Force. A primary review of this study was conducted by HED (see Attachment). The following is a summary of the study and its findings.

The purposes of the study were twofold: 1) determine the amount of residue transferred from a treated vinyl or carpet flooring to the bare hand after an application of a formulation containing deltamethrin (DTM) at different time intervals up to 336 hours after application, and 2) compare residue transfer from the bare hand press procedure with an alternative method for residue transfer, the indoor fogger.

Four Simulated Residential Rooms (SRRs) were used. One room contained the sprayboom apparatus and treated vinyl and carpet flooring. Two other rooms were used to store the treated carpet and vinyl flooring sections for the roller press and one room was used for the hand press procedure. Application of the test material to the flooring was made using a sprayboom apparatus on six different days using 6 different formulations. The desired deposition rate of the test material onto the vinyl and carpet flooring was  $0.9 \mu\text{g}/\text{cm}^2$ . Total deposition was measured using deposition coupons, which were collected after application of the test material followed by a drying period.

For the hand press procedure, test subjects performed one hand press on a separate treated surface at each sampling interval and for the indoor roller procedure; an indoor roller assembly was fitted to the platforms to collect the roller samples.

The study author reported that deposition ranged from  $0.0498$  to  $1.64 \mu\text{g}/\text{cm}^2$ . HED did not correct residue data for field fortification since all average recoveries were above 90%. The average deposition value for each type of run was used in the percent transferability (percent of application) calculations. The validated LOQ reported by the author for each matrix were:  $5.00 \mu\text{g}/\text{sample}$  for alpha cellulose,  $0.100 \mu\text{g}/2$  sponges for dressing sponges, and  $0.100 \mu\text{g}/\text{coupon}$  for percale.

Residues remaining on hands and percale following contact with either a treated vinyl or carpet floor surface were determined from between 4 and 336 hours after the application of DTM. The percent of residue transferred from vinyl to the hands after application was reported from 2.0% (4 hours after application) to <0.1% (72 hours after application). The percent of residue transferred from carpet to the hands after application was reported from 2.23% (4 hours after application) to <0.28% (336 hours after application). The percent of residue transferred from vinyl to percale after application was reported from 0.46% (4 hours after application) to <0.1% (48 hours after application). The percent of residue transferred from carpet to percale after application was reported from 3.26% (4 hours after application) to 0.69% (336 hours after application).

HED also calculated the percent of residue transferred from vinyl flooring and carpet to bare hands and percale. The percent of residue transferred from vinyl to the hands ranged from 3.64% (4 hours after application) to 0.06% (72 hours after application). Residue transferred from carpet to the hands was calculated by HED and ranged from 4.52% (4 hours after application) to 0.11% (336 hours after application). Residue transferred from vinyl to the percale was calculated by HED and ranged from 0.74% (4 hours after application) to 0.08% (48 hours after application). Residue transferred from carpet to the percale was calculated by HED and ranged from 4.02% (4 hours after application) to 0.18% (168 hours after application).

The analysis of the alpha cellulose deposition coupons for the roller and hand presses show that the mean deposition rate for DTM is fairly consistent from application to application and is reproducible. For both hands and percale, the data shows the percent transferability decreasing over time for both carpet and vinyl.

The protocol provided with the study along with OPPTS Series 875 Part B, Guideline 875.2300: Indoor Surface Residue Dissipation, Postapplication and Part C Guidelines were used to review the study. Overall, the majority of the procedures performed and the quality of the data generated in this study conformed to the criteria set forth in the protocol and guidelines.

<b>Summary of Field Fortification Recoveries</b>		
<b>Matrix (µg)</b>	<b>Overall Avg. Recovery (%)</b>	<b>Std. Dev.</b>
	<b>DTM</b>	<b>DTM</b>
Alpha Cellulose	92.4	5.3
Dressing Sponges UV	97.1	6.1
Dressing Sponges MS/MS	93.8	11.6
Percale	90.3	11.8

<b>Deposition of DTM – Results of Alpha Cellulose Coupons</b>		
<b>Sample</b>	<b>DTM Residue (µg/cm<sup>2</sup>)</b>	<b>Standard Deviation</b>
Mean Deposition for Vinyl Roller	0.88	0.15
Mean Deposition for Carpet Roller	0.97	0.20
Mean Deposition for Vinyl Hands	0.93	0.15
Mean Deposition for Carpet Hands	1.27	0.18

\* Deposition (µg/cm<sup>2</sup>) = Individual DTM residues found on alpha cellulose coupons (µg/sample)/surface area of coupon (57.8 cm<sup>2</sup>).

Percale Residue Summary		
Carpet	Time Interval (hrs.)	Percent Transferability
	4	3.26
	8	2.40
	12	2.90
	24	1.13
	48	1.93
	72	1.38
	168	0.40
	336	0.69
Vinyl	Time Interval (hrs.)	Percent Transferability
	4	0.47
	8	0.49
	12	0.27
	24	0.19
	48**	<0.1
	72**	<0.1
	168**	<0.1

Notes:

\*\* Included samples with residues reported to be below the LOQ; used 1/2 the LOQ in calculations (DTM µg/sample).

Hand Residue Summary		
Carpet	Time Interval (hrs.)	Percent Transferability
	4	2.23
	8	1.80
	12	1.44
	24	1.06
	48	0.49
	72	0.28
	168	2.23
	336	1.80
Vinyl	Time Interval (hrs.)	Percent Transferability
	4	1.98
	8	0.52
	12	0.22
	24**	0.07
	48**	0.07
	72**	0.07

Notes:

\*\* Included samples with residues reported to be below the LOQ; used 1/2 the LOQ in calculations (DTM µg/sample).



## UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

WASHINGTON, D.C. 20460

OFFICE OF  
PREVENTION, PESTICIDES  
AND TOXIC SUBSTANCES

Date: September 21, 2007

### **MEMORANDUM**

**SUBJECT:** Study Review: "Measurement of Transfer of Deltamethrin Residues from Vinyl and Carpet Flooring Treated with a Fogger Formulation as a Function of Time" (MRID#: 462976-03)

**FROM:** Zaida Figueroa, Industrial Hygienist  
Registration Action Branch 2  
Health Effects Division (7509P)

**THROUGH:** Jeff Evans, Chair  
Science Advisory Council for Exposure  
Health Effects Division (7509P)

**TO:** Cathryn O'Connell,  
Special Review and Reregistration Division (7508P)

This report reviews a study entitled "Measurement of Transfer of Deltamethrin Residues from Vinyl and Carpet Flooring Treated with a Fogger Formulation as a Function of Time." The protocol provided with the study along with OPPTS Series 875 Part B, Guideline 875.2300: Indoor Surface Residue Dissipation, Postapplication and Part C Guidelines were used to review the study.

**STUDY TYPE:** Active Transfer; Vinyl & Carpet, Transferability over time (roller and hand press)

**TEST MATERIAL:** Deltamethrin; pre-fill batch formulation (similar to an indoor fogger formulation)

**SYNONYMS:** Deltamethrin (DTM)

**CITATION:**

Study Director/Author(s):	Sami Selim, Ph.D.
Title:	Measurement of Transfer of Deltamethrin Residues from Vinyl and Carpet Flooring Treated with a Fogger Formulation as a Function of Time
Study Completion Date:	June 3, 2004
Testing Facility:	Toxcon Health Sciences Research Centre Inc. 9607 - 41st Avenue Edmonton, Alberta Canada T6E 5XL
Analytical Facility:	Enviro-Test Laboratories/XENOS Division Unit 13 - 210 Colonnade Road Nepean, Ontario Canada K2E 7L5
Identifying Codes:	Toxcon Study No.: 02-032-PY01 Xenos Project No.: XEN03-18

**SPONSOR:** Non-Dietary Exposure Task Force

## **EXECUTIVE SUMMARY:**

This report reviews “Measurement of Transfer of Deltamethrin Residues from Vinyl and Carpet Flooring Treated with a Fogger Formulation as a Function of Time” submitted by the Non-Dietary Exposure Task Force. The purposes of the study were: 1) determine the amount of residue transferred from a treated vinyl or carpet flooring to the bare hand after an application of a formulation containing deltamethrin (DTM) at different time intervals up to 336 hours after application, and 2) compare residue transfer from the bare hand press procedure with an alternative method for residue transfer, the indoor fogger.

Four Simulated Residential Rooms (SRRs) were used. One room contained the sprayboom apparatus and treated vinyl and carpet flooring. Two other rooms were used to store the treated carpet and vinyl flooring sections for the roller press and one room was used for the hand press procedure. Vinyl flooring or carpet sections were pinned onto a sheet of plastic-covered plywood attached to the top of six 40 in x 40 in wooden platforms. Application of the test material to the flooring was made using a sprayboom apparatus on six different days using 6 different formulations. The desired deposition rate of the test material onto the vinyl and carpet flooring was  $0.9 \mu\text{g}/\text{cm}^2$ . Total deposition was measured using deposition coupons, which were

collected after application of the test material followed by a drying period. After collection of the deposition coupons, carpet and vinyl flooring sections were removed and moved to their separate rooms. For the hand press procedure, test subjects performed one hand press on a separate treated surface at each sampling interval and for the indoor roller procedure; an indoor roller assembly was fitted to the platforms to collect the roller samples. The subjects' hands were cleaned with isopropyl alcohol dressing sponges to remove any remaining residues after each hand press. The dressing sponges, deposition coupons, and percale roller samples were extracted and then analyzed using a HPLC and HPLC/MS/MS system.

The study author reported that deposition ranged from 0.0498 to 1.64  $\mu\text{g}/\text{cm}^2$ . HED did not correct residue data for field fortification since all average recoveries were above 90%. The average deposition value for each type of run was used in the percent transferability (percent of application) calculations. The validated LOQ reported by the author for each matrix were: 5.00  $\mu\text{g}/\text{sample}$  for alpha cellulose, 0.100  $\mu\text{g}/2$  sponges for dressing sponges, and 0.100  $\mu\text{g}/\text{coupon}$  for percale.

According to the study author, average residues transferred from vinyl flooring to the hand ranged from a high of 0.0185  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to below the LOQ at 72 hours after application. Residues transferred from carpet to hands ranged from a high of 0.0284  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of 0.0036  $\mu\text{g}/\text{cm}^2$  at 336 hours after application. HED calculated hand residues transferred from vinyl flooring and carpet using the raw data provided in the Study Report and used  $\frac{1}{2}$  the LOQ for values reported to be below the LOQ. Residues transferred from vinyl to the hands ranged from a high of 0.034  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of 0.00054  $\mu\text{g}/\text{cm}^2$  at 72 hours after application. Residues transferred from carpet to hands ranged from a high of 0.0574  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of 0.0014  $\mu\text{g}/\text{cm}^2$  at 336 hours after application.

According to the study author, average residues transferred from vinyl to percale ranged from a high of 0.0041  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to below the LOQ at 48 hours after application. Residues transferred from carpet to percale ranged from a high of 0.0317  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of 0.0067  $\mu\text{g}/\text{cm}^2$  at 336 hours after application. HED calculated residues transferred from vinyl and carpet to percale using the raw data provided in the Study Report and used  $\frac{1}{2}$  the LOQ for values reported to be below the LOQ. Residues transferred from vinyl to percale ranged from a high of 0.0065  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of  $2.0 \times 10^{-5}$   $\mu\text{g}/\text{cm}^2$  at 48 hours after application. Residues transferred from carpet to percale ranged from a high of 0.0391  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of 0.0017  $\mu\text{g}/\text{cm}^2$  at 168 hours after application.

The percent of residue transferred from vinyl to the hands after application (based on mean residue) was reported by the study author to range from 2.0% at 4 hours after application to <0.1% at 72 hours after application. The percent of residue transferred from carpet to the hands after application (based on mean residue) was reported by the study author to range from 2.23% at 4 hours after application to 0.28% at 336 hours after application. The percent of residue transferred from vinyl to percale after application (based on mean residue) was reported by the study author to range from 0.46% at 4 hours after application to <0.1% at 48 hours after application. The percent of residue transferred from carpet to percale after application (based on

mean residue) was reported by the study author to range from 3.26% at 4 hours after application to 0.69% at 336 hours after application.

HED also calculated the percent of residue transferred from vinyl flooring and carpet to bare hands and percale. The percent of residue transferred from vinyl to the hands ranged from 3.64% at 4 hours after application to 0.06% at 72 hours after application. Residue transferred from carpet to the hands was calculated by HED and ranged from 4.52% at 4 hours after application to 0.11% at 336 hours after application. Residue transferred from vinyl to the percale was calculated by HED and ranged from 0.74% at 4 hours after application to <0.1% at 48 hours after application. Residue transferred from carpet to the percale was calculated by HED and ranged from 4.02% at 4 hours after application to 0.18% at 168 hours after application.

The protocol provided with the study along with OPPTS Series 875 Part B, Guideline 875.2300: Indoor Surface Residue Dissipation, Postapplication and Part C Guidelines were used to review the study. Overall, the majority of the procedures performed and the quality of the data generated in this study conformed to the criteria set forth in the protocol and guidelines.

#### **COMPLIANCE:**

Signed and dated GLP, Data Confidentiality and Quality Assurance statements were provided. The Study Report noted that the study was performed according to the U.S. EPA FIFRA Good Laboratory Practice Regulations currently in effect (40 CFR, Part 160), with certain exceptions: (1) conventional and digital photography was not done according to GLP Regulations and the resulting photographs are considered non-GLP, (2) scanning of hand palmar surface area to create a digital image and the computerized method of calculating surface area was not done according to GLP Regulations, and (3) information recorded on subject entry, exit and hand inspection forms was not entered and/or corrected according to GLP Regulations.

#### **GUIDELINE OR PROTOCOL FOLLOWED:**

The study was conducted following Xenos and Toxcon Standard Operating Procedures and the protocol of the Non-Dietary Exposure Task Force (Toxcon Study No. 02-032-PY01).



## **I. MATERIALS AND METHODS**

### **A. Materials:**

#### **1. Test Material:**

Formulation: An unidentified pre-fill formulation similar to what is used for an indoor fogger; developed by Bayer Environmental Science as an oil phase product containing 1.29% w/w deltamethrin. The oil phase formulation was then diluted to form the test substance (pre-fill emulsion; deltamethrin 0.15% (wt/wt)).

Lot/Batch #: Batch Number s: 0305-1, 0305-2, 0305-3

Formulation guarantee: Certificate of analyses provided.

CAS #(s): 52918-63-5

Other Relevant Information: Toxcon ID No.: PY01T016

#### **2. Relevance of Test Material to Proposed Formulation:**

Deltamethrin is an active ingredient used in formulated consumer products intended for use in human dwellings to control ants, fleas, and other domestic insects. The product used was a pre-fill batch formulation similar to what is used for an indoor fogger formulation developed by Bayer Environmental Science. The name and label for the test product was not provided with the study.

### **B. Study Design:**

There were five deviations and one amendment from the study protocol. The amendment included: (1) the company addresses for the study director and the quality assurance unit because both individuals relocated their businesses.

The deviations from the protocol included (1) the field fortifications and blanks were prepared two days prior to the first sprayboom application for vinyl; (2) the reference substance was kept in a freezer instead of in a refrigerator (Protocol Section 9.2.2) (3) deposition coupons C and D were missing on the vinyl hand press spray day; (4) the platforms were 66" from back wall instead of 68" as specified in figure 1 of the protocol; and (5) the deposition coupons were not placed exactly as illustrated in Figure 3, except for the roller spray day. It was noted whether the deviations had any impact on the study. The Study Report noted that the Method XAM-70 was modified as follows: (1) Section 6.2 was modified to indicate the calibration solutions were prepared in acetonitrile:water/1:1; (2) Section 7.3.1 was modified to indicate that extracts of alpha cellulose were reconstituted first in acetonitrile instead of methanol, the final volume was also made up with acetonitrile:water/1:1; and (3) Section 7.5.3 was modified to indicate that extracts of dressing sponges and percale were reconstituted first in acetonitrile instead of methanol, the final volume was also made up with acetonitrile:water/1:1.

## 1. Site Description:

Test locations:	The test site was located at the Toxcon Health Sciences Research Centre in Canada. Four test rooms (Simulated Residential Rooms (SRRs)) were used with one containing the application equipment (the sprayboom). The rooms were prepared according to Toxcon SOP No. E-025: Preparation of Test Rooms Prior to an Experiment.
Meteorological Data:	Target test room conditions prior to application included an air exchange rate of $0.6 \pm 0.1$ air change per hour (ACH), a temperature of $72 \pm 4^{\circ}\text{F}$ and a relative humidity of $50 \pm 10\%$ .
Ventilation/Air-Filtration:	The ventilation system for the application room was turned off (dampers closed) during application and for three hours after application. After the three hours, the dampers were opened for a 30-minute drying period and the room conditions were adjusted to reach the conditions prior to application.

## 2. Surface(s) Monitored:

Room(s) Monitored:	Four SRRs were used. One room contained the sprayboom apparatus and treated vinyl and carpet flooring. Two other rooms were used to store the treated carpet and vinyl flooring sections for the roller press and one room was used for the hand press procedure.
Room Size(s):	16 ft x 16 ft x 8 ft
Types of Surface(s):	Vinyl and carpet flooring
Surface Characteristics:	Vinyl and carpet flooring sections were pinned onto a sheet of plastic-covered plywood attached to the top of six 40 in x 40 in wooden platforms. The carpet and vinyl flooring specifications were provided in the protocol. Each platform included 4 deposition coupons and 11 flooring sections.
Areas sprayed and sampled:	The carpet and vinyl flooring sections in one of the three SRRs used in this study were sprayed and sampled for DTM residues. Prior to the application of the test substance using the sprayboom equipment, the platforms were rolled together and placed to the center of the room.
Other products used:	N/A

## 3. Physical State of Formulation as Applied: Fogger

#### 4. Application Rates and Regimes:

Application Equipment: Sprayboom

Application Regime: Six sprayboom runs (conducted on separate days) were conducted in one SRR. The runs included two for the vinyl roller procedure, two for the carpet roller procedure, one for the carpet hand press procedure and one for the vinyl hand press procedure.

Application rate(s): An application rate was not provided in the Study Report. Application was based on the desired deposition rate of the test material onto the vinyl and carpet flooring. The desired deposition rate was  $0.9 \mu\text{g}/\text{cm}^2$ . Deposition rate was based on the results of indoor DTM total release fogger deposition study. The sprayboom nozzle sweep speed required to obtain the desired deposition was calculated using the following equation:  $U = [(Q)(F_a)(k_1)]/[(R)(n)(d)(10^{-6})]$ , where U is the sprayboom nozzle sweep speed (cm/s), Q is the nozzle output rate (g/s),  $F_a$  is the fraction of deltamethrin in the formulation, R is the target deposition rate of DTM ( $\mu\text{g}/\text{cm}^2$ ), d is a fixed value representing the distance between nozzles (71.2 cm), n is the number of nozzles (5), and  $k_1$  is a correction factor to account for formulation that is sprayed, but not deposited, on the test surface. The target speed was not provided in the Study Report but was reported to be documented in the raw data.

Equipment Calibration Procedures: The Study Report states that a calibrated sprayboom was used in the study, but calibration procedures were not provided. It is not certain if the equipment used in this study was consistent with the proposed use for this product. A label was not provided with the study. Therefore, the label recommended application method is not known.

Was total deposition measured? Total deposition was measured using deposition coupons. The deposition coupons consisted of squares of alpha cellulose (3 in x 3 in). The coupons were backed with hexane-wiped heavy duty aluminum foil. The Study Report states that coupons were prepared according to Toxcon SOP No. M-015: Preparation of Alpha Cellulose Deposition Coupon. The coupons were present on the wooden platforms during test substance application.

### C. Sampling:

Surface Areas Sampled:	Vinyl and carpet flooring sections present on wooden platforms in SRR.
Replicates per sampling interval:	<p>Twelve subjects participated in the study. Hand presses were performed with both the left and right hand of the test subjects. Each subject performed one hand press on a treated section of vinyl flooring at 4, 12, 24, 72, 168 and 336 hours after application. Hand presses were performed on carpet at the same time intervals as the vinyl flooring. Each hand press used a new section of treated vinyl or carpet flooring, resulting in a total of 10 hand press replicates for both vinyl and carpet per time interval. Transferability of pesticides to the bare hand was determined as a function of pre-specified applied force (8 kg) and contact duration (20 seconds).</p> <p>For the indoor roller, two sprayboom runs were performed and triplicate samples of percale for each flooring type were taken at 4, 8, 12, 24, 48, 72, 168 and 336 hours after application.</p>
Number of sampling intervals:	For hand press samples, 6 sampling intervals were conducted for carpet and vinyl flooring. For roller samples, 8 sampling intervals were conducted for carpet and vinyl flooring.
Method and Equipment:	Residue deposition was determined using alpha cellulose deposition coupons and transfer of residue to hands and percale was determined using hand presses and isopropyl alcohol dressing sponge wipes, and percale indoor rollers.
Sampling Procedure(s):	
Deposition coupons -	The deposition coupons were collected following a drying period after application of the test material (approximately 3.5 hours after application). Disposable latex gloves were worn when the coupons were handled. The coupons were folded, so that the exposed side was on the inside, and then wrapped in hexane-wiped aluminum foil.
Hand residues -	After application and collection of the deposition coupons, vinyl flooring and carpet sections were removed and moved to a hand press room. Each section of the carpet and vinyl flooring was placed in a hand press balance configuration at specific sampling intervals. The transfer of residues was

determined based on the applied force (~8 kg) and contact duration (~20 s). The subjects washed and dried their hands prior to the hand presses. After the hand presses, the subjects' hands were cleaned with isopropyl alcohol wetted hand wipes (dressing sponges). Hand palmer surface areas were determined using an ink image of the palm side of each hand, which was then scanned into a computer to create a digital image of the hand. The computerized methods of calculating surface areas are described in Toxcon SOP No. M-010.

Indoor rollers -

Percal was used as the test material for the indoor roller dosimeter. The Study Report stated that the design and use of the indoor rollers was described in Toxcon SOP M-011. The indoor roller assembly was fitted to the platforms to collect the roller samples. After each use of the roller, the frame assembly was wiped according to Toxcon SOP M-011.

Sample Handling and Storage:

The hand wipes from each hand were placed in separate pre-labeled 180 mL glass jars with Teflon lined lids. Deposition coupons and percale samples were placed in aluminum containers and moved to freezer storage (<-5°C) within 3 hours of collection. Samples were shipped to the analytical laboratory overnight in an insulated cooler with dry ice.

#### IV. ANALYTICAL METHODOLOGIES

##### A. Extraction method:

Samples were analyzed using Xenos Analytical Method XAM-70. Extraction was performed by sonication and mechanical shaking of the dressing sponges at room temperature with ethyl acetate. For the alpha cellulose coupons, the total extracts were taken to dryness by rotary evaporation and for the percale samples and the dressing sponges, an aliquot was taken to dryness using rotary evaporation. Samples of percale coupons and the dressing sponges were reconstituted into hexane, while the alpha cellulose extracts were reconstituted into acetonitrile:water and further diluted for injection on HPLC/UV. Percal extracts were cleaned-up using the Isolute silica SPE procedure. Dressing sponges were partitioned twice, with sodium chloride (20%). Then the hexane portion was partitioned portion with acetonitrile. The acetonitrile portion was taken to dryness, reconstituted and cleaned-up using the Isolute silica SPE procedure. The final volume of cleaned-up extracts was 1:1 acetonitrile:water prior to injection.

**B. Detection methods:** DTM analysis was performed using a HPLC/UV system and for some dressing sponges where co-extracts from the hand of subjects interfere the analysis was performed using a HPLC/MS/MS system. The analysis was validated using both quantification procedures and obtaining comparable results. See Table 1 for specific conditions.

Table 1: HPLC/UV & HPLC/MS/MS Chromatographic Conditions			
HPLC/UV			
HPLC Column	Zorbax SB C8: 150 x 4.6 mm, 3.5 μm Guard: Phenomenex Security Guard C8: 4.0 x 3.0 mm Temperature: 40°C		
Gradient	Time (min)	A (%) ACN	B (%): Caner
	0.01	60	40
	12.0	90	10
	16.0	90	10
	19.0	100	0
	24.0	100	0
	26.0	60	40
Wavelength	230 nm		
AUFS	0.005		
Flow Rate	0.800 mL/min		
Injection Volume	100 μL		
Retention Time	Approximately 12.5 minutes		
HPLC/MS/MS			
HPLC Column	Waters Symmetry Shield RP-8: 100 x 3.0 mm, 3.5 μm Guard: Phenomenex Security Guard C8: 4.0 x 3.0 mm Temperature: 40°C		
Gradient	Time (min)	A (%) ACN	B (%): Caner
	0	30	70
	2.0	30	70
	4.0	10	90
	9.0	10	90
	9.1	30	70
	16.2	30	70
A	25 mM ammonium acetate in water		
B	Acetonitrile		
Flow Rate	0.50 mL/min		
Injection Volume	20 μL		

**C. Method Validation:** The analytical methods were validated in a previous study. The Study Report states that validation data for the limit of quantitation (LOQ) was taken from Xenos report XEN03-18. The LOQ is reported for DTM for each matrix (see Table 2).

Table 2: Validated LOQs	
Matrix	DTM
Alpha cellulose	5.00µg/sample
Dressing sponges	0.100 µg/2 sponges
Percle (121 cm <sup>2</sup> )	0.100 µg/coupon

#### D. Instrument performance and calibration:

Calibration solutions for the HPLC/UV system were prepared monthly by dilution of solution E (10 µg of deltamethrin/mL) with 1:1 acetonitrile:water. The solutions were stored in a freezer at ~ -10°C. For the HPLC/UV the calibration range was 15.0 ng/mL to 150.0 ng/mL. The calibration range for the HPLC/MS/MS system was 4.0 ng/mL to 100.0 ng/mL.

#### E. Quality Control:

- Lab Recovery:** To obtain recovery and method performance data, concurrent laboratory control samples were fortified with the formulated product. In each set extracted, two fortifications were carried out and at least one of those was at the LOQ. The formulated product was applied to the matrix using a syringe. Results from the laboratory fortified samples are summarized in Table 3. Overall average recoveries for the alpha cellulose coupons were  $102.0 \pm 7.4$  (7.3% RSD). Overall average recoveries for the dressing sponges were  $91.8 \pm 10.4$  (11.3 % RSD) for HPLC/UV and  $96.4 \pm 10.9$  (11.3% RSD) for HPLC/MS/MS. Overall average recoveries for the percle coupons were  $93.1 \pm 9.9$  (10.7% RSD).
- Field Fortification:** Samples of the alpha cellulose coupons were fortified at 2xLOQ and 10xLOQ. The dressing sponges were fortified at 2xLOQ and 20xLOQ, and the percle coupons were fortified at 2xLOQ and 5xLOQ. Three to six field fortification samples were prepared at each level for each flooring type. These samples were stored and analyzed with the test samples. Field fortification results are summarized in Table 4. Overall average recoveries for the alpha cellulose coupons were  $92.4 \pm 5.3$  (5.8% RSD). Overall average recoveries for the dressing sponges were  $97.1 \pm 6.1$  (6.3 % RSD) for HPLC/UV and  $93.8 \pm 11.6$  (12.4% RSD) for HPLC/MS/MS. Overall average recoveries for the percle coupons were  $90.3 \pm 11.8$  (13.0% RSD).
- Control Samples:** Field control samples were prepared according to Toxcon SOP M-016. Laboratory control samples were prepared by adding a volume of solvent approximately equal to the largest volume of solution used in the fortifications to samples for each flooring

Storage Stability: Field fortification samples were analyzed after the maximum storage time of experiment samples to verify storage stability. Alpha cellulose samples were analyzed after 72 days, dressing sponges were analyzed after 134 days, and percale samples were analyzed after 151 days. All recoveries verified the stability of the residues.

## **V. RESULTS**

HED did not correct residue data for field fortification since recoveries were above 90%.

### **A. Alpha Cellulose and Deposition of Formulation:**

The average deposition DTM reported by the study author for each sprayboom run is provided in Table 5. There were 6 different sprayboom runs using 3 different formulations. Deposition ranged from 0.957 to 1.27  $\mu\text{g}/\text{cm}^2$ . The average deposition value for each type of run was used in the percent transferability (percent of application) calculations (see Table 5).

### **B. Hand Residues:**

Residues transferred to bare hands from carpet were calculated by the study author for each hand of the test subjects at 4, 12, 24, 72, 168, and 336 hours after application and residues transferred to bare hands from vinyl were calculated at 4, 12, 24, 72, 168 and 336 hours after application. Residues were reported only if residues were above the LOQ. According to the study author, DTM average residues transferred from vinyl to the hand ranged from a high of 0.0185  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to <LOQ at 72 hours after application. DTM residues transferred from carpet to hands ranged from 0.0284  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of 0.0036  $\mu\text{g}/\text{cm}^2$  at 336 hours after application.

HED calculated hand residues transferred from vinyl and carpet using the raw data provided in the Study Report and used  $\frac{1}{2}$  the LOQ for values reported to be below the LOQ. DTM residues transferred from vinyl to the hands ranged from a high of 0.034  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of 0.00054  $\mu\text{g}/\text{cm}^2$  at 72 hours after application. DTM residues transferred from carpet to hands ranged from a high of 0.0574  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of 0.0014  $\mu\text{g}/\text{cm}^2$  at 336 hours after application.

The percent of residue transferred to the hands after contact with either treated vinyl or carpet surfaces was calculated as the ratio of the amount of residue present on the hand divided by the average residue found on the alpha cellulose coupons for that particular sprayboom run. The average residue found on the coupons for the vinyl-hand press runs was reported to be 0.933  $\mu\text{g}/\text{cm}^2$ . The average residue found on the coupons for the carpet-hand press runs was reported to be 1.27  $\mu\text{g}/\text{cm}^2$ .



The percent of residue transferred from vinyl to the hands after application was reported by the study author to range from 2.0% at 4 hours after application to <0.1% at 72 hours after application. The percent of residue transferred from carpet to the hands after application was reported by the study author to range from 2.23% at 4 hours after application to 0.28% at 336 hours after application. The percent of residue transferred from vinyl to the hands calculated by HED ranged from 3.64% at 4 hours after application to 0.06% at 72 hours after application. Residue transferred from carpet to the hands was calculated by HED and ranged from 4.52% at 4 hours after application to 0.11% at 336 hours after application.

### **C. Percal Roller Residues:**

Residues transferred to percale from carpet were calculated by the study author at 4, 8, 12, 24, 48, 72, 168, and 336 hours after application and residues transferred to percale from vinyl were calculated at 4, 8, 12, 24, 48, 72, 168 and 336 hours after application. Residues were reported for DTM only if residues were above the LOQ. According to the study author, DTM residues transferred from vinyl to percale ranged from a high of 0.0041  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to <LOQ at 48 hours after application. DTM residues transferred from carpet to percale ranged from a high of 0.0317  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of 0.0067  $\mu\text{g}/\text{cm}^2$  at 336 hours after application.

HED calculated percale residues transferred from vinyl and carpet using the raw data provided in the Study Report and used  $\frac{1}{2}$  the LOQ for values reported to be below the LOQ. DTM residues transferred from vinyl to percale ranged from a high of 0.0065  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of  $2.0 \times 10^{-5}$   $\mu\text{g}/\text{cm}^2$  at 48 hours after application. Residues transferred from carpet to percale ranged from a high of 0.0391  $\mu\text{g}/\text{cm}^2$  at 4 hours after application to a low of 0.0017  $\mu\text{g}/\text{cm}^2$  at 168 hours after application.

The percent of residue transferred to percale after contact with either treated vinyl or carpet surfaces was calculated as the ratio of the amount of residue present on the percale divided by the average residue found on the alpha cellulose coupons for that particular sprayboom run. The average residue found on the coupons for the vinyl-roller runs was reported to be 0.880  $\mu\text{g}/\text{cm}^2$ . The average residue found on the coupons for the carpet-roller runs was reported to be 0.973  $\mu\text{g}/\text{cm}^2$ .

The percent of residue transferred from vinyl to percale after application was reported by the study author to range from 0.46% at 4 hours after application to <0.1% at 48 hours after application. The percent of residue transferred from carpet to percale after application was reported by the study author to range from 3.26% at 4 hours after application to 0.69% at 336 hours after application. Residue transferred from vinyl to the percale was calculated by HED and ranged from 0.74% at 4 hours after application to 0.08% at 48 hours after application. Residue transferred from carpet to the percale was calculated by HED and ranged from 4.02% at 4 hours after application to 0.18% at 168 hours after application.

## **VI. CONCLUSION**

Residues remaining on hands and percale following contact with either a treated vinyl or carpet flooring surface were determined from 4 hours after application to 336 hours after application. The percent of residue transferred from vinyl to the hands after application was reported by the study author to range from 2.0% at 4 hours after application to <0.1% at 72 hours after application. The percent of residue transferred from carpet to the hands after application was reported by the study author to range from 2.23% at 4 hours after application to 0.28% at 336 hours after application. The percent of residue transferred from vinyl to percale after application was reported by the study author to range from 0.46% at 4 hours after application to <0.1% at 48 hours after application. The percent of residue transferred from carpet to percale after application was reported by the study author to range from 3.26% at 4 hours after application to 0.69% at 336 hours after application.

HED also calculated the percent of residue transferred from vinyl flooring and carpet to bare hands and percale. The percent of residue transferred from vinyl to the hands ranged from 3.64% at 4 hours after application to 0.06% at 72 hours after application. Residue transferred from carpet to the hands was calculated by HED and ranged from 4.52% at 4 hours after application to 0.11% at 336 hours after application. Residue transferred from vinyl to the percale was calculated by HED and ranged from 0.74% at 4 hours after application to 0.08% at 48 hours after application. Residue transferred from carpet to the percale was calculated by HED and ranged from 4.02% at 4 hours after application to 0.18% at 168 hours after application.

## **VII. LIMITATIONS OF THE STUDY**

The protocol provided with the study along with OPPTS Series 875 Part B, Guideline 875.2300: Indoor Surface Residue Dissipation, Postapplication and Part C Guidelines were used to review the study. Overall, the majority of the procedures performed and the quality of the data generated in this study conformed to the criteria set forth in the protocol and guidelines.

**Table 3: Summary of Laboratory Fortification Recoveries**

Matrix (µg)	Average Fortification Level (ug)	Average Measured Residue (µg/sample)	Average Recovery (%)	Std. Dev.	Overall Average Recovery (%)	Std. Dev.	% RSD
	DTM	DTM	DTM	DTM	DTM	DTM	DTM
Alpha Cellulose	LOQ				102.2	7.4	7.3
	5.0	4.9	98.9	6.7			
	5xLOQ						
	25.0	26.4	105.7	11.3			
	10xLOQ						
	50.0	53.2	106.3	7.9			
	20xLOQ						
	100.0	104.8	104.8	3.64			
Dressing Sponges UV	LOQ				91.8	10.4	11.3
	0.1	0.1	89.6	11.9			
	10xLOQ						
	1.0	1.0	95.6	7.6			
	50xLOQ						
	5.0	4.5	89.9	9.72			
Dressing Sponges MS/MS	LOQ				96.4	10.9	11.3
	0.1	0.1	95.3	10.7			
	10xLOQ						
	1.0	1.0	99.6	10.2			
	50xLOQ						
	5.0	4.6	91.6	11.3			
Percale	LOQ				93.1	9.9	10.7
	0.1	0.1	85.3	22.4			
	5xLOQ						
	0.5	0.4	72.3	36.5			
	10xLOQ						

	1.0	1.0	96.1	7.5			
	20xLOQ						
	2.0	2.06	103.0	5.66			
	50xLOQ						
	5.0	4.9	98.6	--			

\* Samples not reported or with an amount found (µg/sample) equal to zero were not used in calculating fortification level averages or the overall average recovery.

Table 4: Summary of Field Fortification Recoveries

Matrix (µg)	Average Fortification Level (ug)	Average Measured Residue (µg/sample)	Average Recovery (%)	Std. Dev.	Overall Average Recovery (%)	Std. Dev.	% RSD
	DTM	DTM	DTM	DTM	DTM	DTM	DTM
Alpha Cellulose		~2XLOQ					
	9.815	9.5	96.9	3.6	92.4	5.3	5.8
		~10xLOQ					
	49.05	43.1	87.9	1.3			
Dressing Sponges UV		~2XLOQ					
	0.197	0.2	100.4	6.3	97.1	6.1	6.3
		~20XLOQ					
	1.970	1.8	93.8	4.1			
Dressing Sponges MS/MS		~2XLOQ					
	0.197	0.1	103.6	3.1	93.8	11.6	12.4
		~20XLOQ					
	1.970	1.8	88.9	11.2			
Percale		~2XLOQ					
	3.450	3.1	90.8	8.7	90.3	11.8	13.0
		~5XLOQ					
	8.375	7.5	89.7	15.1			

Table 5: Deposition of DTM - Results of Alpha Cellulose Coupons			
Application Date	Sample	Residue ( $\mu\text{g}/\text{cm}^2$ )	Standard Deviation
		DTM	DTM
5/28/2003	Vinyl Roller Run #1	0.96	0.14
5/29/2003	Vinyl Roller Run #2	0.80	0.16
6/3/2003	Carpet Roller Run #1	0.92	0.20
6/5/2003	Carpet Roller Run #2	1.02	0.20
5/31/2003	Vinyl Hand Press	0.93	0.15
6/7/2003	Carpet Hand Press	1.27	0.18
Mean Deposition for Vinyl Roller		0.88	0.15
Mean Deposition for Carpet Roller		0.97	0.20
Mean Deposition for Vinyl Hands		0.93	0.15
Mean Deposition for Carpet Hands		1.27	0.18

\* Deposition ( $\mu\text{g}/\text{cm}^2$ ) = DTM residues found on alpha cellulose coupons ( $\mu\text{g}/\text{sample}$ )/surface area of coupon ( $57.8 \text{ cm}^2$ ).

Table 6: Summary of Deltamethrin Hand Residues from Contact with Treated Carpet and Vinyl Flooring							
Time Interval (hours)	Average Residue (µg/sample) *	Surface Area (cm²)	Average Residue (ng/cm²) ***	Avg. Residue Std Deviation	Deposition Coupons (µg/cm²)	% of Application	% Application Std Deviation
Carpet							
4	2.20	79.2	28.37	11.04	1.270	2.23	0.92
12	1.74		22.92	7.98		1.80	0.66
24	1.43		18.30	3.33		1.44	0.28
72	1.05		13.44	2.29		1.06	0.19
168	0.49		6.25	1.12		0.49	0.09
336	0.27		3.60	1.47		0.28	0.12
Vinyl							
4	1.35	74.09	18.51	7.74	0.933	1.98	0.83
8	0.35		4.87	2.09		0.52	0.22
12	0.15		2.04	1.18		0.22	0.13
24**	0.05		0.69	0.11		0.07	0.01
48**	0.05		0.69	0.11		0.07	0.01
72**	0.05		78.27***	0.66		0.12	0.07

\* Average of 10 replicates (5 subjects using both hands) for all carpet and vinyl hand residues intervals.

\*\* Included samples with residues reported to be below the LOQ; used 1/2 the LOQ in calculations (DTM µg/sample).

\*\*\* Average residue (ng/cm<sup>2</sup>) calculated by taking individual replicate residue (µg/sample) and multiplying by individual replicate hand surface area provided in the Study Report.

\*\*\*\* Two subjects from the previous sampling interval were substituted for two new subjects. The hand surface area (cm<sup>2</sup>) values for the two new subjects were included to this sampling interval for a total of 10 replicates.

Table 7: Summary of Deltamethrin Percal Residues from Contact with Treated Carpet and Vinyl Flooring							
Time Interval (hours)	Average Residue (µg/sample) *	Surface Area (cm <sup>2</sup> )	Average Residue (ng/cm <sup>2</sup> ) ***	Avg. Residue Std Deviation	Deposition Coupons (µg/cm <sup>2</sup> )	% of Application	% Application Std Deviation
Carpet							
4	88.4	2787.0	31.73	6.65	0.974	3.26	0.68
8	65.2		23.38	3.09		2.40	0.32
12	78.7		28.23	6.41		2.90	0.66
24	30.7		11.00	2.84		1.13	0.29
48	52.4		18.81	3.56		1.93	0.37
72	37.5		13.44	3.20		1.38	0.33
168	10.8		3.86	1.97		0.40	0.20
336	18.6		6.69	0.61		0.69	0.06
Vinyl							
4	11.40	2787.0	4.10	2.08	0.880	0.47	0.24
8	12.10		4.33	1.36		0.49	0.15
12	6.51		2.33	0.50		0.27	0.06
24	4.66		1.67	0.42		0.19	0.05
48**	0.05		0.02	0.00		0.00	0.00
72**	0.05		0.02	0.00		0.00	0.00
168**	0.05		0.02	0.00		0.00	0.00

\* Included average of three replicates for all carpet and vinyl roller time intervals.

\*\* Included samples with residues reported to be below the LOQ; used 1/2 the LOQ in calculations (DTM= 0.100 µg/sample)

\*\*\* Average residue calculated (ng/cm<sup>2</sup>) by taking individual replicate residue (µg/sample) and dividing by individual surface area provided in the Study Report.



## **APPENDIX A**

**Compliance Checklist for "*Measurements of Transfer of Deltamethrin Residue from Vinyl and Carpet Flooring Treated with a Fogger Formulation as a Function of Time*"**

**Compliance Checklist for "Measurements of Transfer of Deltamethrin Residue from Vinyl and Carpet Flooring Treated with a Fogger Formulation as a Function of Time"**

**GUIDELINE 875.2300  
INDOOR SURFACE RESIDUE DISSIPATION  
POSTAPPLICATION**

1. *The test substance must be the typical end use product of the active ingredient.* This criterion was met. The formulation is similar to products used in residences.
2. *The production of metabolites, breakdown products, or the presence of contaminants of potential toxicologic concern, should be considered on a case-by-case basis.* This criterion does not apply to this study. There was no mention of metabolites, breakdown products or other contaminants.
3. *Indoor surface residue studies should be conducted under ambient conditions similar to those encountered during the intended use season, and should represent reasonable worst case conditions.* This criterion was met.
4. *Ambient conditions (i.e., temperature, barometric pressure, ventilation) should be monitored.* This criterion was met. Target conditions were identified and apparently met.
5. *The end use product should be applied by the application method recommended on the label. Information that verifies that the application equipment (e.g., sprayer) was properly calibrated should be included.* These criteria do not apply.
6. *The application rate used in the study should be provided and should be the maximum rate specified on the label. However, monitoring following application at a typical application rate is more appropriate in certain cases.* This criterion was met. Although an application rate was not provided in the Study Report, the application was based on the desired deposition rate of the test material onto the vinyl and carpet flooring.
7. *If multiple applications are made, the minimum allowable interval between applications should be used.* This criterion does not apply to this study.
8. *Indoor surface residue (ISR) data should be collected from several different types of media (e.g., carpeting, hard surface flooring, counter tops, or other relevant materials).* This criterion was met; the indoor surface residue (ISR) data was collected from two different types of media, vinyl and carpet flooring.

9. *Sampling should be sufficient to characterize the dissipation mechanisms of the compound (e.g., three half-lives or 72 hours after application, unless the compound has been found to fully dissipate in less time; for more persistent pesticides, longer sampling periods may be necessary). Sampling intervals may be relatively short in the beginning and lengthen as the study progresses. Background samples should be collected before application of the test substance occurs.* This criterion was met. The sampling was made at different time intervals up to 336 hours after application.
10. *Triplicate, randomly collected samples should be collected at each sampling interval for each surface type.* This was met.
11. *Samples should be collected using a suitable methodology (e.g., California Cloth Roller, Polyurethane Roller, Drag Sled, Coupons, Wipe Samples, Hand Press, vacuum cleaners for dust and debris, etc.) for indoor surfaces.* This criterion was met.
12. *Surface sampling should be conducted in conjunction with air sampling. Enough duplicate air samples should be taken in a room to establish a dissipation curve.* This criterion does not apply.
13. *Samples should be stored in a manner that will minimize deterioration and loss of analytes between collection and analysis. Information on storage stability should be provided.* This criterion was met. A separate storage stability study was not performed; however, field fortification samples were stored for the maximum storage time and recoveries were found to be acceptable.
14. *Validated analytical methods of sufficient sensitivity are needed. Information on method efficiency (residue recovery), and limit of quantitation (LOQ) should be provided.* This criterion was met.
15. *Information on recovery samples must be included in the study report. A complete set of field recoveries should consist of at least one blank control sample and three or more each of a low-level and high-level fortification. These fortifications should be in the range of anticipated residue levels in the field study.* This criterion was met.
16. *Raw residue data must be corrected if appropriate recovery values are less than 90 percent.* This criterion was met.
17. *Indoor surface residues should be reported as mg per m<sup>2</sup> or cm<sup>2</sup> of surface sampled. Distributional data should be reported, to the extent possible.* This criterion was met.
18. *Reported residue dissipation data in conjunction with toxicity data should be sufficient to support the determination of a reentry interval.* This criterion does not apply.